

# Development of a Novel Continuous Processing Technology for Functionally Graded Composite Energetic Materials using an Inverse Design Procedure

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## I. Introduction

For a variety of applications, the functional requirement of a material can vary with location within a structure. One way to address this has been to use different materials, joined together so as to take care of the functional requirements at different locations. This unfortunately gives rise to undesirably high stress concentrations at the interface, when the structure is loaded, which might lead to failure. Attempts at controlling these stresses have led to the concept of Functionally Graded Materials (FGMs). FGMs are structures that possess gradual variations in material behavior that enhance material and/or structural performance [1]. For example, at one point the material may be hard and at another point it may be soft. The description of this functional variation is known as the FGM architecture. Typical architectural parameters include layer thickness,  $t$ , and composition gradient,  $p$ . In designing FGMs, it is desirable to determine the architectural parameters that optimize system performance for a given application by modeling the relationship between the processing of a FGM, the microstructures that develop, and their related properties.

FGMs are being applied to a variety of structural and nonstructural applications. Recently, FGM concepts have become of interest to the U.S. Navy to improve large caliber gun propellant performance by replacing a 7-perf grain with a single perf grain that has the same performance, but burns more efficiently because it possesses a functionally graded architecture. In the case of composite energetic materials used as solid rocket propellants, referred to as a *grain*, the volume fraction of ingredients, such as 30 and 200 micron AP particles ( $V_{AP}$ ) can be varied along the length of the grain to produce a corresponding difference in burn rate. It can be noted that the burn rate is related not only the volume fraction of AP particles, but the particle size distribution as well. One of the technical challenges to develop functionally graded solid rocket propellants is the lack of a design methodology and manufacturing technology for processing continuously graded architectures. Therefore, it is necessary to develop a novel continuous processing technology for FGMs using an inverse design procedure that can be applied to propellants.

A novel continuous processing technology that has shown a great deal of promise for solid rocket propellants is known as Twin Screw Extrusion (TSE) [2]. TSE processing can be applied not only to functionally grading solid rocket propellants, but to FGMs in general. For solid rocket propellants, it will be desirable to tailor the burn rate performance in a monolithic rocket motor utilizing new design and control schemes for TSE based on the continuously graded microstructures that can be achieved in

this novel process. This will require the development of an inverse design procedure that will ultimately serve as a tool for the designers and manufacturers of solid rocket motors. The in-flight performance of a rocket motor (simply that part of a rocket consisting of propellant, its case, and a nozzle) will be first specified in the inverse design procedure. Employing the inverse design procedure, the graded microstructure that achieves the specified performance can be predicted, and the manufacturing control parameters for producing it are also dictated.

In the inverse design procedure, component design and fabrication are synergistically combined, not just for the manufacturing of FGMs but for the establishment of an entirely new approach to engineering structures [3]. An objective function is used to define specimen performance by a designer, and then the inverse fabrication problem is solved to obtain the gradient microstructures that satisfy the objective function. Models of the evolution of gradient microstructures during the fabrication process are essential to solving the inverse problem. These models will depend on the ingredients of the materials, which are selected from a database of available materials that can potentially satisfy the design requirements for the given application (e.g., composite energetic materials for solid rocket propellants). The optimal design can therefore be determined from a diverse selection of materials with a variety of gradient architectures. This complicated design problem necessitates the use of robust design evaluation techniques, such as optimization methods, to determine the optimal design.

These complications are simplified by assuming that the materials for the design have already been chosen and that the functional gradient in the system is only along one direction. This reduces the number of design variables in the optimization problem, as well as the size of the design space. Various optimization methods are available to arrive at the solution that achieves the best value for the objective. Given the inherent complexity of describing the physical properties and performance of gradient microstructures, the Genetic Algorithm technique has proven to be a very robust technique to achieve the global optimum.

Finally, the performance functionally graded architecture must be assessed to determine the objective function for a given gradient microstructure. For functionally graded solid rocket propellants, microstructural models of burning rate performance are employed. These performance models are integrated with processing models and optimization methods to complete the inverse design procedure.

This paper elucidates on the development of a novel continuous processing technology for functionally graded

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composite energetic materials (FGCEMs) using an inverse design procedure as follows:

1. Description of continuous processing of FGCEMs for solid rocket motor applications
2. Description of the Inverse Design Procedure
3. Use of the Twin Screw Extrusion process to fabricate functionally graded composite energetic materials
4. Process models to describe the evolution of gradient microstructures
5. Characterization of gradient microstructures
6. Modeling of burn rate performance
7. Optimization of the gradient microstructure using the Genetic Algorithm

## II. Continuous Processing of FGCEMs for Solid Rocket Motor Applications

While composite energetic materials for rocket motor applications are typically processed homogeneously using batch techniques, the novel continuous processing technology known as Twin Screw Extrusion (TSE) has been recently demonstrated to produce higher quality products. However, the true potential of the continuous processing aspects of the technology have yet to be fully realized. In addition to process safety, economy, flexibility, and quality, the most interesting feature of TSE is the ability to produce continuously graded microstructures. These graded microstructures have a significant impact on direct concepts for solid rocket motor applications.

Conventional solid rocket motors consist of a uniform propellant composition throughout. They are typically produced this way on purpose, necessitated by safety and performance considerations. The consequence of uniform composition is uniformity in burning rate behavior, and thus the thrust performance is very predictable. This results in a constraint on design concepts for rocket motors, where the only design variables becomes the topological characteristics of the motor. Ideal rockets should have a controllable thrust, which is true for liquid propellants where the fuel is metered to the combustion chamber. However, liquid propellants are not well suited for many rocket motor applications due to higher needs for storage and handling safety and shelf-life limitations among others.

There is a desire for solid rocket motors that can exhibit more than one burning rate profile while in flight. The answer to this is a functionally graded rocket propellant. Thinking of the propellant as a long cylinder, the composition would be graded in the length direction (*Figure 1*). For an end-burning type rocket, there would be one propellant formulation, identified as composition A, at the ignition with a particular burning characteristic. At some point during flight, there would be a point of transition to a second type of propellant, identified as composition B, with a different burning characteristic that was specified by the designer to maximize the rocket's potential to reach its target. As shown in *Figure 3* the volume of composition B at one end is zero and 100 percent at the other. At some point between the two, there is a continuous and smooth transition from one to the other.

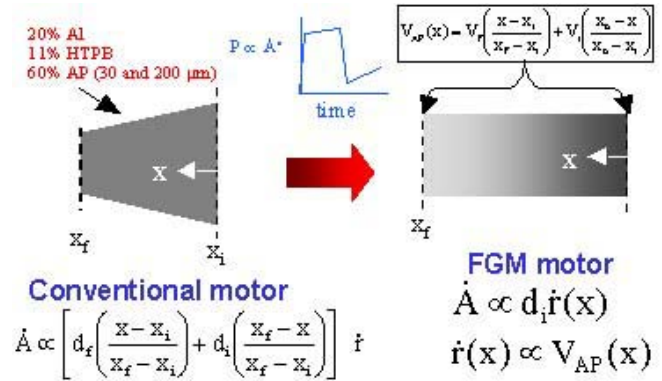


Figure 1. Comparison of traditional solid rocket motor concept with FGM concept

The TSE process is naturally suited to produce this type of gradient from one composition to another. Operating a twin screw extruder at one steady condition and dynamically changing the ingredients to produce a new formulation will result in the extrudate changing from the original composition to second one. Because of the inherent backmixing in a twin screw extruder, an abrupt change in ingredients results in a more gradual change in the composition of the extruded product. There are several ways to achieve this for a rocket propellant. One simple idea is to decrease the volume of oxidizer that reduces the burn rate. However a more effective idea is to change the ratio of coarse to fine sized particles. A combination of the two coupled with a process change, such as extruder screw rpm, will be studied. In this way, the continuous process is especially well suited to producing functionally graded materials. In contrast the batch process is incapable of producing a smooth and continuous transition.

In addition to variations in the ingredients, the operating conditions of the TSE can also be varied. This can be used to control the extent and composition profile of the graded microstructure. It will also be possible to vary the screw configuration of the extruder to alter the characteristics of the graded microstructure. The effects of dynamic variations in ingredients and operating conditions for a given screw configuration must be modeled in order to predict the evolution of graded microstructures in the TSE process using the inverse design procedure.

## III. Inverse Design Procedure for FGCEMs

The inverse design procedure can be summarized as follows:

1. Define the required target performance or the system objectives
2. Select the material components for the graded architecture
3. Evaluate the material structure and its properties from various relationships.
4. Evaluate the optimal gradient of the materials using Genetic Algorithm.
5. Fabricate the optimal FGM through processes like twin-screw extrusion.
6. Test the fabricated FGM for the required properties

7. If the required properties are not achieved, either choose different components for the FGM architecture or redefine the optimization parameter.

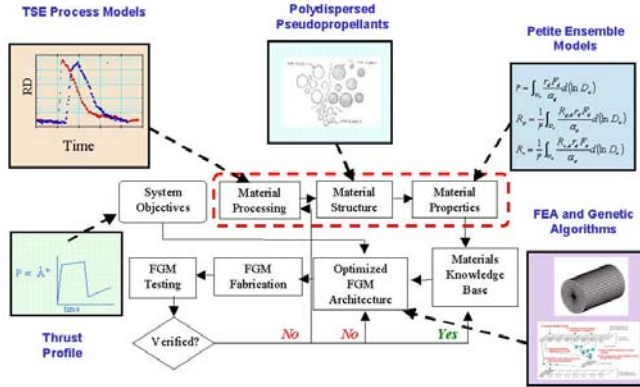


Figure 2. Flowchart showing the Inverse Design Procedure for FGCEMs

In the present discussion, we are interested in the description of the design and fabrication of FGCEMs. It is assumed that the ingredients for the energetic composite material are given, so the material knowledge base is limited to this ingredient formulation. Therefore, the focus of the inverse design procedure is on integrating process and performance models with the Genetic Algorithm optimization method.

#### IV. Twin Screw Extrusion Processing of Graded Microstructures

TSE processes are utilized to manufacture a world of consumer and industrial goods from snack foods and medical tubing to plastic pellets and military propellants. The process is a continuous type in that the twin screw extruder will produce a product as long as the ingredient supply is maintained. Because this type of process has so many advantages over batch type, it has found widespread utility across diverse industries. For most however the advantages are universal: economy, quality, environmental, flexibility, and safety. In the case of energetic materials, all these advantages have been proven. Some of these illustrate why the twin screw extruder shows great promise for producing functionally graded materials.

To understand the TSE process, a description of the equipment is necessary. The extruder consists of two screws, typically fully intermeshing, which run through temperature-controlled barrels. The barrels are modular in design and specialized for feeding solid and liquid ingredients, vacuum, or other functions. They are interchangeable allowing a configuration best suited to a particular process. Furthermore the screws consist of various segmented elements that slide onto the screw shafts allowing for customizable screw designs. Like the barrel sections, various screw geometries are available which are utilized for conveying ingredients, gentle and high shear mixing, devolatilization, and many others. Hence the mixer is highly configurable and thus very flexible allowing for the optimization of many types of processes. In less than a work

shift, the mixer can be reconfigured for a completely different product.

This flexibility lends itself to facility expenditure savings. As with the case of naval gun propellants, the discrete batch process required a number of individual process steps, each with its own facility, equipment and operators. The continuous process with a twin screw extruder eliminates some steps and allows the rest to be combined. The result is a great reduction in the number of facilities required and one continuous operation. In that the mixing/extrusion/cutting operation is remotely operated, the safety of the process is much greater by reduced operator exposure to hazards and reduced quantities in a mixing state at any point in time.

Furthermore the continuous process yields a more consistent product thus improving overall quality. There is significantly less variation in material, and the efficiency of mixing is better than batch methods. The process lends itself to on-line analysis allowing for the quick detection of anomalous conditions or material automatically diverting it to waste. In the batch process this is impossible until later in the production after a large quantity of potentially bad product has been made.

The extruder is only the heart of the process (Figure 3). The process is supported by various ingredient feeders. The quality of the extruded product is directly influenced by the accuracy of the ingredient addition. For this reason only the most accurate feeding technologies are utilized. These commonly include loss-in-weight control for solid ingredients and flow metering control for liquids.

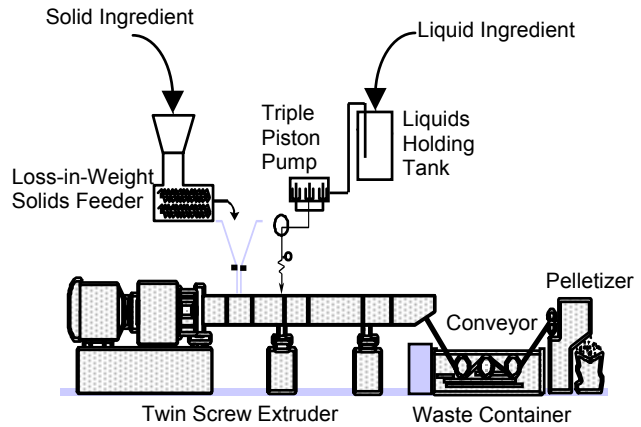


Figure3. Twin-screw extrusion process

Other support equipment includes product collection. For the example shown in Figure 3, the extruded strand is conveyed to a pelletizer where it is cut by a rotating blade into short *pellets*. Not shown are the temperature control units for the extruder barrels and the process control system for the facility.

As mentioned previously, changing the ingredients, operating conditions, and screw configuration can affect the evolution of gradient microstructures. This is demonstrated in Figure 4.



Figure 4. Graded Composites manufactured using the TSE process

## V. Modeling of TSE Process

Several inert trials have yielded good indications of proof-of-principle thus demonstrating the concept. There are a number of research issues to understand the nature of the process that will ultimately lead to a predictive model. These include quantitative material characterization to study the coupling between the process and the microstructure in order to develop appropriate process models.

Material transport through the TSE is accomplished by screw geometry and screw motion [4]. The geometry is highly flexible in that screw elements of different shapes can be added together in different combinations. Screws that have been removed from the barrels immediately after a mid-operation stop can be seen in Figure 5. Note the differences in geometry in regards to pitch, direction, and shape.

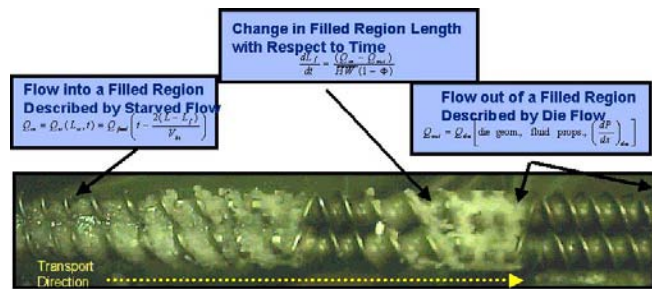


Figure 5. Picture of Unmixed and Mixed Material on Twin Screws after Process Halt and Extraction

The direction of flow through the barrels is from the left to the right in the Figure. This is a good illustration of the state, mixing and distribution of material in the process at any given time. In general, twin screw extruders are starve fed, i.e., the screw channels aren't completely full in the locations where ingredients are added. However the degree of fill in the screws downstream of ingredient addition is variable depending upon the geometry, ingredient throughput rate, screw rpm, and time. Places of high fill, including 100 percent, occur in sections where there are mixing and left-handed screw elements. There is material too on the conveying screws, though the degree of fill is significantly less.

The characterization of TSE processes are often described using Resident Time Distributions (RTDs) [5]. RTDs provide a measure of the time over which the material that is fed in the extruder will reside in the extruder as follows:

$$e(t) = \frac{c(t)}{\int_0^{\infty} c(t') dt'} \quad [1]$$

where  $e(t)$  is the RTD and  $c(t)$  is a filtered probe response obtained from the extruder. These resident times are directly related to the flow of the material within the extruder. The residence time distribution is a characteristic of the process that is studied in order to quantify the dampening as a result of backmixing that occurs in the extruder. Under normal and steady operating conditions there is a continuous supply of material conveyed to the mixing zones, and an equal amount conveyed away. Much of the literature for the twin screw process is characterization of the steady state. Using a 1-D conservation of mass model of the TSE process, the dynamic RTD can be used to describe the gradient in composition that evolves in the TSE. This 1-D model is also illustrated in Figure 5.

## VI. Characterization of Gradient Microstructures

To verify the gradient microstructures predicted by the process model, the gradient microstructure must be characterized. This requires the development of techniques for quantifying the gradient microstructure in polymer composites with very high solids loading (<90%) and multimodal particle distributions. Generally homogeneity is highly desirable for any engineered product, and characterization methods have evolved to describe homogeneous products and deviations from homogeneity such as poor dispersion, voids, cracks, impurities, agglomerations, etc. FGMs by nature have designed microstructures that change with location, and therefore are non-homogeneous. Yet the nonhomogeneity is still ordered. The characterization techniques must be quantitative, objective, and repeatable.

Quantitative techniques are most important to the research on FGMs. Common techniques include SEM and optical stereological analysis as well as various physical property measurements [6-8]. Preliminary characterization work indicates that the most promising method may be optical stereoscopy coupled with digital image analysis. Since the microstructure of an FGM varies with position, serial sectioning and sampling are necessary. The biggest challenge for highly filled polymer-bonded composites, i.e., solid rocket propellant, is particle loss (or pull-out) during cutting and polishing. The combination of hard and soft phases presents significant challenges. Improvement of the surface is necessary to reveal the microstructure. However the more steps in the process and the longer the step, then the better the appearance of the filler at the expense of increasing filler loss. Figure 6 illustrates the effect of surface improvement and resulting Fourier analysis of the particle distribution.



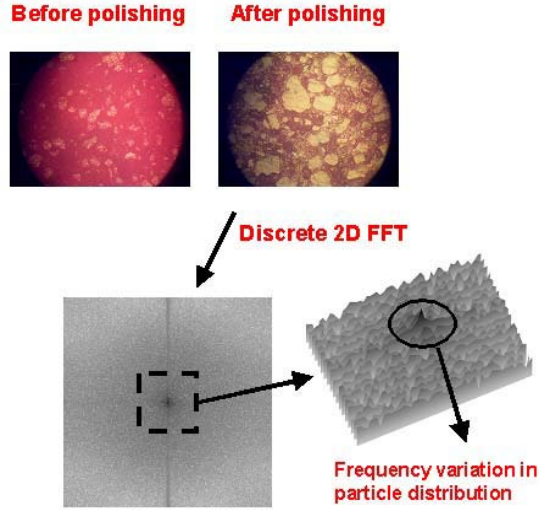


Figure 6. The effects of improving the surface of a cut sample to reveal the microstructure of the TSE-processed particle-reinforced composite, and the resulting 2-D FFT of the microstructure.

## VII. Burn Rate Performance Modeling

For determining the material gradient and distribution in a propellant, a model is required to describe the combustion process. Based on the prediction of this model, the performance of the FGCEM can be optimized. For this investigation a steady-state combustion model called the Petite Ensemble Model (PEM) is used [9]. The PEM is based on a statistical treatment of the propellant surface with multiple flame structure centered about characteristic oxidizer particles. This model can be summarized by the following equations:

$$F_d = \frac{1}{(2\pi \ln \sigma)^{1/2}} \exp \left[ -\frac{1}{2} \left( \frac{\ln D_o - \ln \bar{D}_o}{\ln \sigma} \right)^2 \right] \quad [2]$$

$$\bar{r} = \int_{D_o} \frac{r_d F_d}{\alpha_d} d(\ln D_o) \quad [3]$$

$$R_p = \frac{1}{\bar{r}} \int_{D_o} \frac{R_{p,d} r_d F_d}{\alpha_d} d(\ln D_o) \quad [4]$$

$$R_v = \frac{1}{\bar{r}} \int_{D_o} \frac{R_{v,d} r_d F_d}{\alpha_d} d(\ln D_o) \quad [5]$$

where  $F_d$  is the overall oxidizer distribution function,  $D_o$  is the oxidizer particle diameter,  $\bar{D}_o$  is the mean oxidizer particle diameter,  $\sigma$  is the oxidizer distribution function mode width parameter,  $\bar{r}$  is the composite propellant mean burning rate,  $r_d$  is the burn rate for a pseudopropellant,  $\alpha_d$  is the pseudopropellant oxidizer mass fraction,  $R_p$  is the composite propellant pressure coupled response function,  $R_{p,d}$  is the pseudopropellant pressure coupled response function,  $R_v$  is the composite propellant velocity coupled response function, and  $R_{v,d}$  is the pseudopropellant velocity coupled response function. PEM predictions of the variation in burning rate with composition for TSE processed energetic materials can be seen in Figure 7.

Since the combustion process has a propensity for interacting with the pressure and velocity fields in the combustion chamber, their effects have to be accounted for in the model. This is especially important for the graded microstructures, where there is constant variation in these fields. To achieve these effects in the graded microstructure, a small perturbation is applied to the steady-state PEM equations to yield non steady-state models of both pressure and velocity coupled response of composite solid propellants. The main parameter that describes the performance of a rocket motor is the pressure variation with time in the engine. From this the required burn rate of the propellant can be found if the material gradient and the combustion mechanisms are known. A Fortran program is used to characterize the propellant burn properties with the input of the material distribution and the initial conditions based on the Petite Ensemble Model. While the 1-D graded architectures can be analyzed directly, Finite Element Models (FEMs) have to be employed to describe this variation for 2-D and 3-D graded architectures.

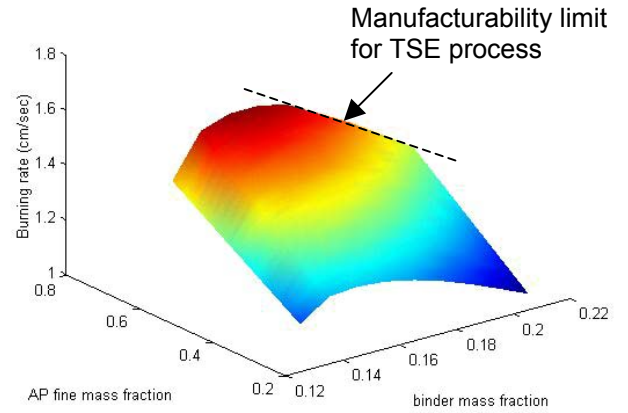


Figure 7. PEM prediction of variation in burning rate with composition for TSE processed energetic materials

## VIII. Optimization of Gradient Microstructures using Genetic Algorithm Optimization Technique

Genetic algorithms can be used for searching complex solution spaces for an optimal solution. The genetic algorithm consists of three progressive steps and an iterative step as follows:

1. Define an objective function.
2. Generate a initial random population of solutions.
3. Evaluate the performance of the population using the objective function, apply reproduction, crossover and mutation to the old population to generate the children from the parents.
4. Repeat step 3 until the convergence criteria is reached.

The GA used to optimize the graded material distribution employs a Differential Evolution strategy, which is an advanced form of GA (Figure 8). It is comprised of three sub-programs.

1. **Definition of the objective function:** The objective function, whose minimum is to be found out, is defined in this section. This could incorporate the finite element analysis of the system to obtain the parameters required to evaluate the objective function.
2. **Declaration of initial values:** Various parameters like the number of population members, targeted value of the objective function, initial guesses of the values of the variables, range of values for the population members, optimization strategy to be used, minimum and maximum number of iterations etc. are specified in this routine. This routine calls the main part of the program, which is described below.
3. **Differential Evolution routine:** This routine uses different strategies to generate a set of children from the parent population and evaluates their "fitness" or the value of the defined objective function. Since in this case our goal is to minimize the value of the objective function, the fitness of a member is based on how low the evaluated objective function value is. This procedure is repeated till a population member is obtained that gives the minimum objective function value.

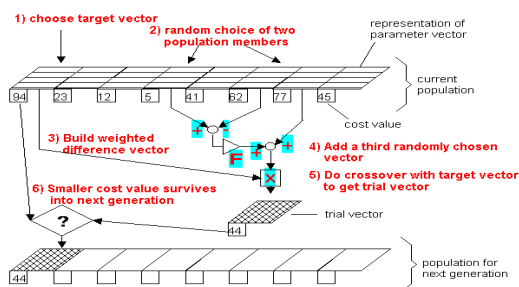


Figure 8. Differential Evolution Genetic Algorithm used to optimize functionally graded solid rocket propellants

To utilize this GA, the minimum value of the objective function should be known beforehand to detect convergence. Although the GA will converge to the vicinity of the optimum faster, it may take many more iterations to reach the absolute minimum. One drawback of this GA is the inability to constrain the search space. However, it is possible to penalize the objective function to confine the search. Recently, it has been demonstrated that the efficiency of the GA can be enhanced using the graded architectures [10]. This makes it feasible to integrate the process and performance models with the optimization method in the inverse procedure for the developing the novel continuous processing technology to fabricate functionally graded energetic materials.

## IX. Conclusions

A novel continuous processing technology is described for functionally graded composite energetic materials using an inverse design method. The continuous processing technology is based on Twin Screw Extrusion (TSE), and the inverse design method is used to integrate a burning rate performance model with a model of the TSE processing effects on the evolution of the gradient microstructure to determine the optimal gradient microstructure using mathematical optimization methods, such as Genetic Algorithms.

The research requires efforts in three scientific areas: processing science, materials characterization, and computational modeling. The combination of these efforts provides models and tools for the inverse design procedure. Using the TSE process to produce functionally graded materials requires the description of dynamic resident time distributions (RTDs) using a 1-D conservation of mass model to account for transient effects on material mixing and transport processes in the extruder. The process models are then used to relate the microstructural evolution of the gradient to the various process controls. Because solid propellant is comprised of an unusual combination of a soft elastomeric matrix that is highly filled with hard inorganic crystals, new techniques for preparing adequate samples for analysis and quantitatively characterizing the microstructure are necessary.

To model the performance of functionally graded solid rocket propellants, a Petite Ensemble Model (PEM) is used. This model can account for dynamic effects from pressure and cross-flow velocity fields that are attributed to the graded microstructure. The processing and performance models are then integrated with a mathematical optimization method known as the Differential Evolution Genetic Algorithm (DEGA) to complete the inverse design procedure for functionally graded solid rocket propellants. Thus, optimal gradient microstructures that can be manufactured using the TSE process for rocket motor applications can be determined.

## X. Acknowledgements

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